National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 3090

Aroclors in Transformer Oil
(SRMs 3075 to 3080)

This Standard Reference Material (SRM) is a set of six different solutions of individual Aroclors in transformer oil and consists of six 2-mL ampoules, each containing approximately 1.2 mL of each of the following SRMs: SRM 3075, Aroclor 1016 in Transformer Oil; SRM 3076, Aroclor 1232 in Transformer Oil; SRM 3077, Aroclor 1242 in Transformer Oil; SRM 3078, Aroclor 1248 in Transformer Oil; SRM 3079, Aroclor 1254 in Transformer Oil; and SRM 3080; Aroclor 1260 in Transformer Oil. This SRM is intended primarily for calibrating chromatographic instrumentation and methods of analysis used for the determination of Aroclors and polychlorinated biphenyls (PCBs) in transformer oil. The SRMs that comprise SRM 3090 are also available individually as a set of five 2-mL ampoules of each Aroclor in transformer oil.

In addition, SRMs are available that consist of a set of five 2-mL ampoules of each Aroclor listed above in methanol (SRM 3081 to 3086) and as a set of six solutions, each containing approximately 1.2 mL of each of the listed Aroclors (SRM 3091).

Certified Concentrations of Aroclors: The certified concentrations [1,2] given below are based on results obtained from the gravimetric preparation of each solution and from the analytical results determined using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

SRM	Type of Aroclor	CAS Registry Number[a]	Concentration (mg/kg)	Concentration (mg/L)
3075	Aroclor 1016	12674-11-2	17.1 ± 1.0	15.2 ± 0.9
3076	Aroclor 1232	11141-16-5	4252 ± 114	3789 ± 106
3077	Aroclor 1242	53469-21-9	4102 ± 87	3656 ± 82
3078	Aroclor 1248	12672-29-6	3658 ± 161	3260 ± 146
3079	Aroclor 1254	11097-69-1	3579 ± 154	3190 ± 139
3080	Aroclor 1260	11096-82-5	1079 ± 98	962 ± 88

[a] Chemical Abstracts Service (CAS) Registry Number

The results are expressed as the certified value ± the expanded uncertainty. The certified values are the unweighted average of the concentrations determined by gravimetric and gas chromatographic measurements. The expanded uncertainties, at the 95% level of confidence, are calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO Guide [3] and $k = 2$ is the coverage factor. The value of u_c includes an allowance for differences between the concentration determined by gas chromatographic measurements for various sources of Aroclors and gravimetric preparation. The concentrations expressed as a volume fraction were obtained by multiplying the certified values, expressed as mass fractions, by the measured density (22 °C) of the SRMs, 0.8912 g/mL ± 0.0205 g/mL, where 0.0205 represents one standard deviation (1s) and is incorporated in the volume fraction uncertainties.

Expiration of Certification: The certification of this SRM lot is valid until **31 July 2012**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 23 May 2003

John Rumble, Jr., Chief
Measurement Services Division

The coordination of the technical measurements leading to the certification was under the direction of D.L. Poster and M.M. Schantz of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by D.L. Poster of the NIST Analytical Chemistry Division.

Preparation of the SRM was performed by M.P. Cronise of the Measurement Services Division and D.L. Poster of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

Partial support for the preparation and certification of this Standard Reference Material was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

NOTICE AND WARNING TO USERS

Handling and Storage: This material contains solutions of PCB mixtures. PCB-containing materials are reported to be toxic. Extreme caution and care should be exercised during the handling of SRM 3090. Contact your regional EPA office for information regarding proper disposal. Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Instructions for Use: Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty. Certified values are not applicable to material stored in ampoules that have been opened for more than 5 minutes, even if they are resealed.

PREPARATION AND ANALYSIS[1]

The Aroclors used in the preparation of this SRM were obtained from the U.S. EPA with the exception of Aroclor 1016 which was obtained from a commercial source. Each oil in SRM 3090 was prepared at NIST by weighing and mixing each Aroclor into transformer oil (Univolt 60, Exxon). The Aroclors were added to transformer oil and mixed until completely dissolved and homogenized. The total mass of each solution was measured and aliquots (1.2 mL) were dispensed into 2-mL amber glass ampoules, which were then flame sealed. Aliquots from nine ampoules of each Aroclor in transformer oil, selected randomly, were analyzed using capillary gas chromatography with electron capture detection and an immobilized non-polar (5% phenyl methylpolysiloxane) stationary phase column. For each solution of Aroclor, an internal standard solution containing chlorinated compounds that were not present in the Aroclor was added to each sample for quantification purposes. Prior to gas chromatography, samples were placed on aminopropyl solid phase extraction columns and eluted with hexane. The concentrated eluants were then placed on a semi-preparative aminopropylsilane column using hexane as the mobile phase. Calibration solutions consisting of weighed amounts of each Aroclor and internal standard compounds in transformer oil were processed alongside the samples and chromatographically analyzed to determine response factors for each Aroclor relative to the internal standards. The results for each Aroclor are based on the areas of the dominant Aroclor PCB peaks relative to the internal standard peaks (Figure 1). This approach is similar to U.S. EPA Method 505 (Analysis of organohalide pesticides and commercial polychlorinated biphenyl (PCB) products in water by microextraction and gas chromatography, revision 2.0).

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

[1] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).

[2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI);* NIST Special Publication 811, U.S. Government Printing Office: Washington, DC (1995).

[3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

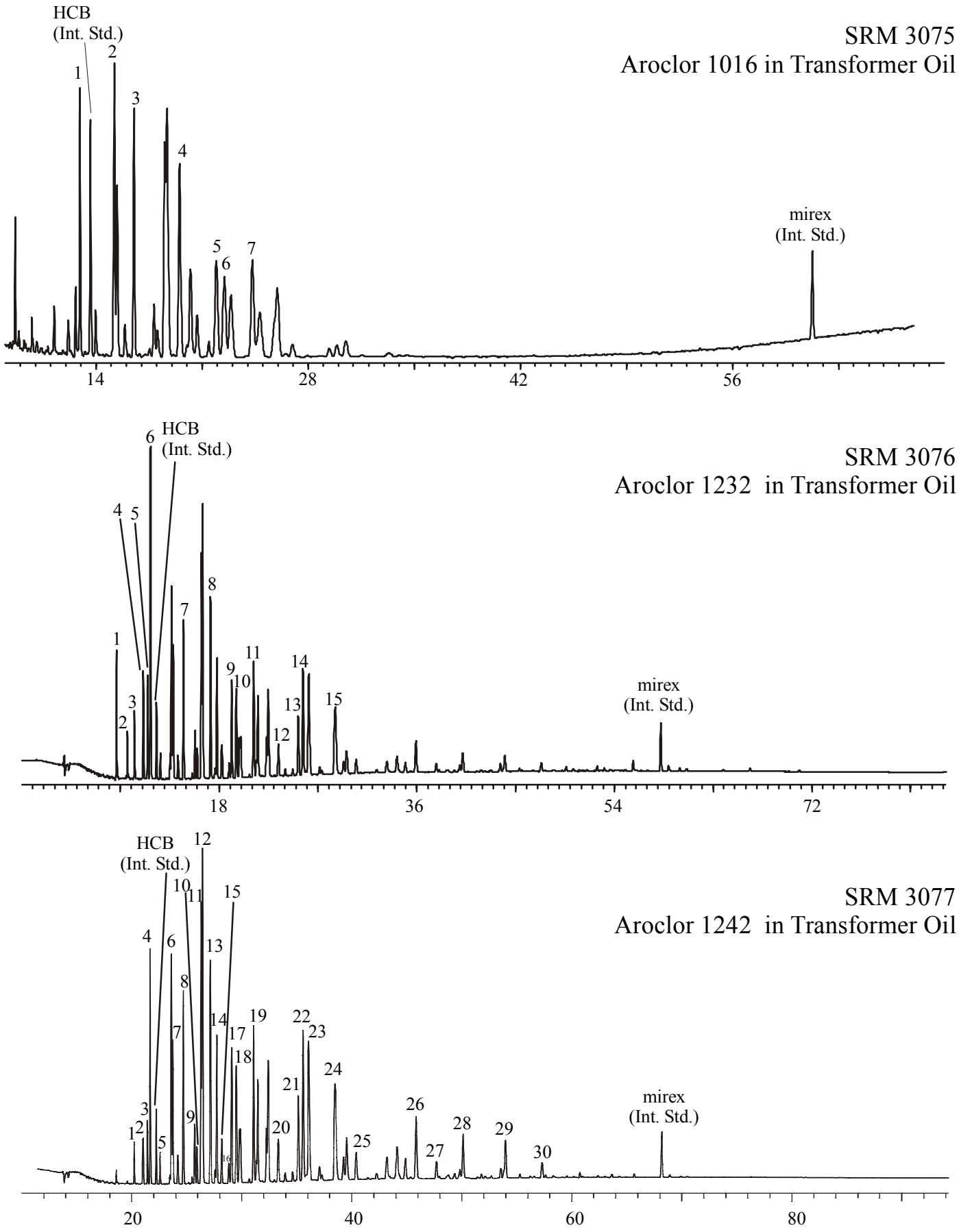
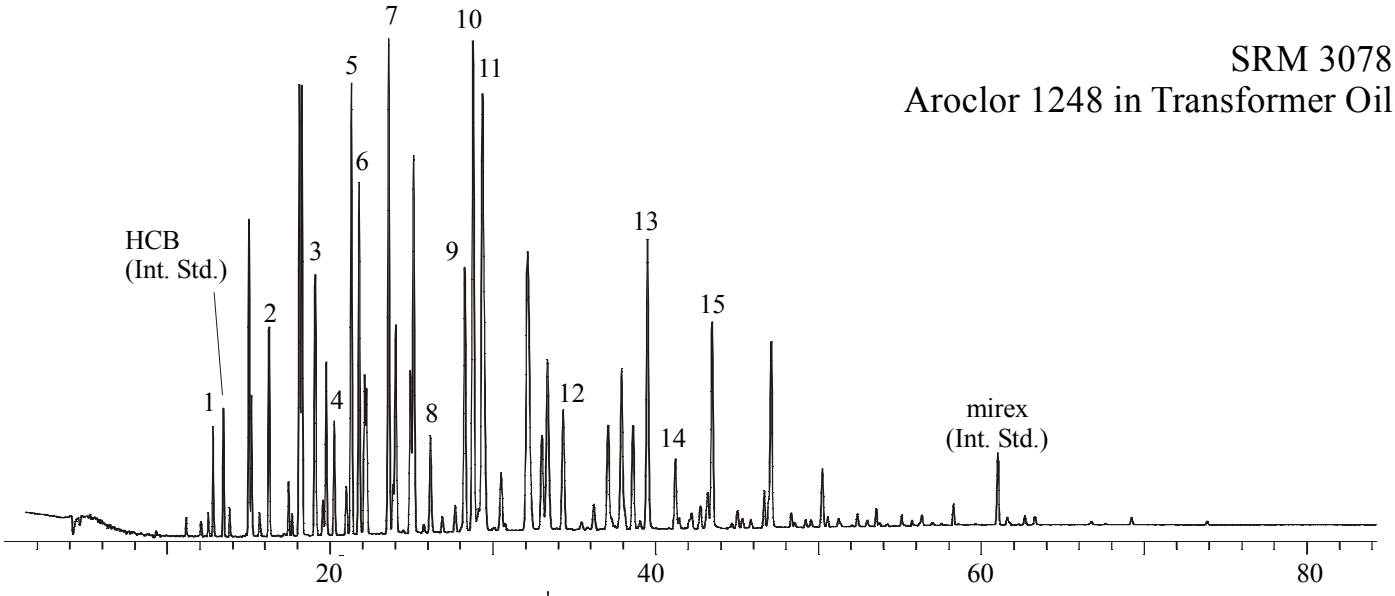
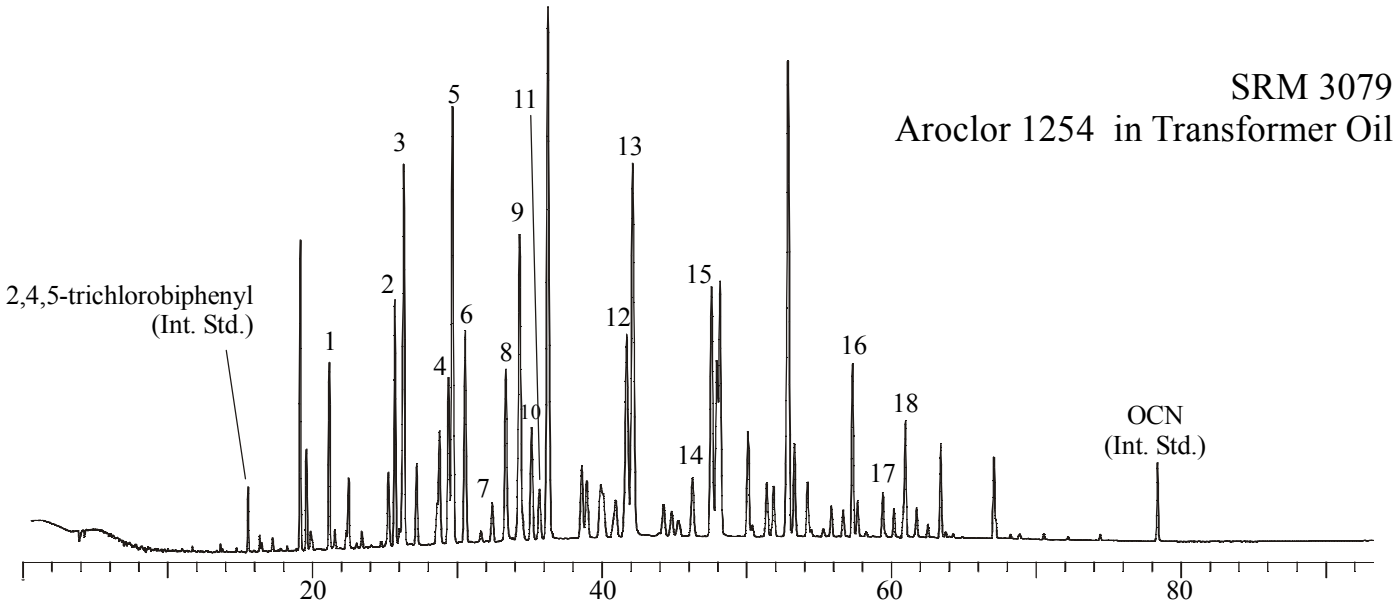


Figure 1. Gas chromatograms from the analysis of Aroclors in transformer oil. The peaks used for quantification of the Aroclor mass in each transformer oil are shown. (Int. Std.) = internal standard, x-axis represents "time" in minutes





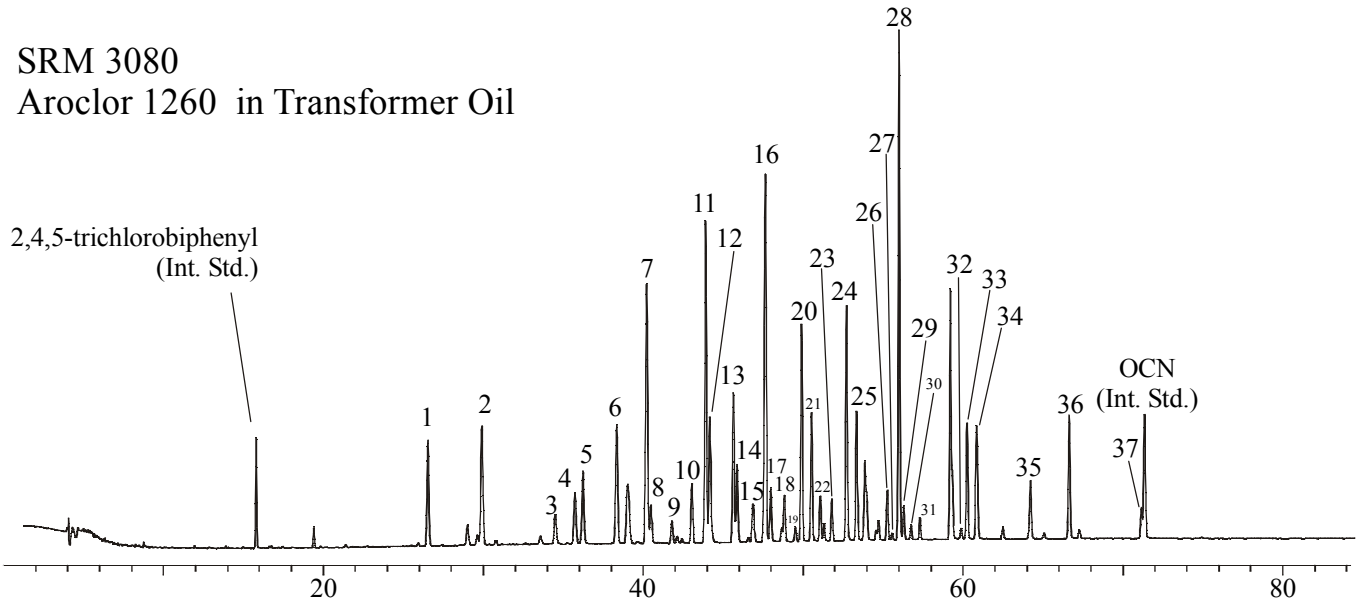


Figure 1, continued. Gas chromatograms from the analysis of Aroclors in transformer oil. The peaks used for quantification of the Aroclor mass in each transformer oil are shown. (Int. Std.) = internal standard, x-axis represents "time" in minutes